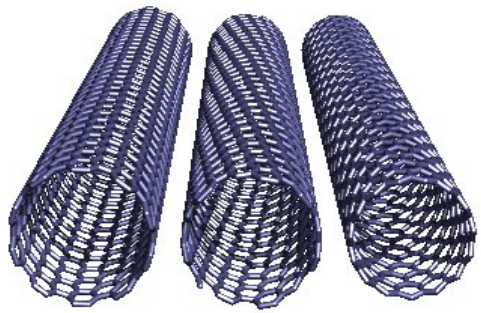
Quality Control of Bulk Single Wall Carbon Nanotube Materials

Ursula Dettlaff



Max-Planck-Institute for Solid State Research, Stuttgart

2nd Joint Workshop on Measurement Issues in Single Wall Carbon Nanotubes: Purity and Dispersion Part II,
January 26-28, 2005, NIST, Gaithersburg, MD, USA

Preliminary Protocol for Quality Control of Bulk Single Wall Carbon Nanotube Materials

Deliverable for EU-Project SPANG
Germany, Austria, Italy, China

Purpose:

to deliver standard analytical instructions for quality control which can be used independently in each research group of the consortium getting the same results

Also used by runnig EU Projects: CARDECOM, CANAPE
7 european countries with 15 research groups

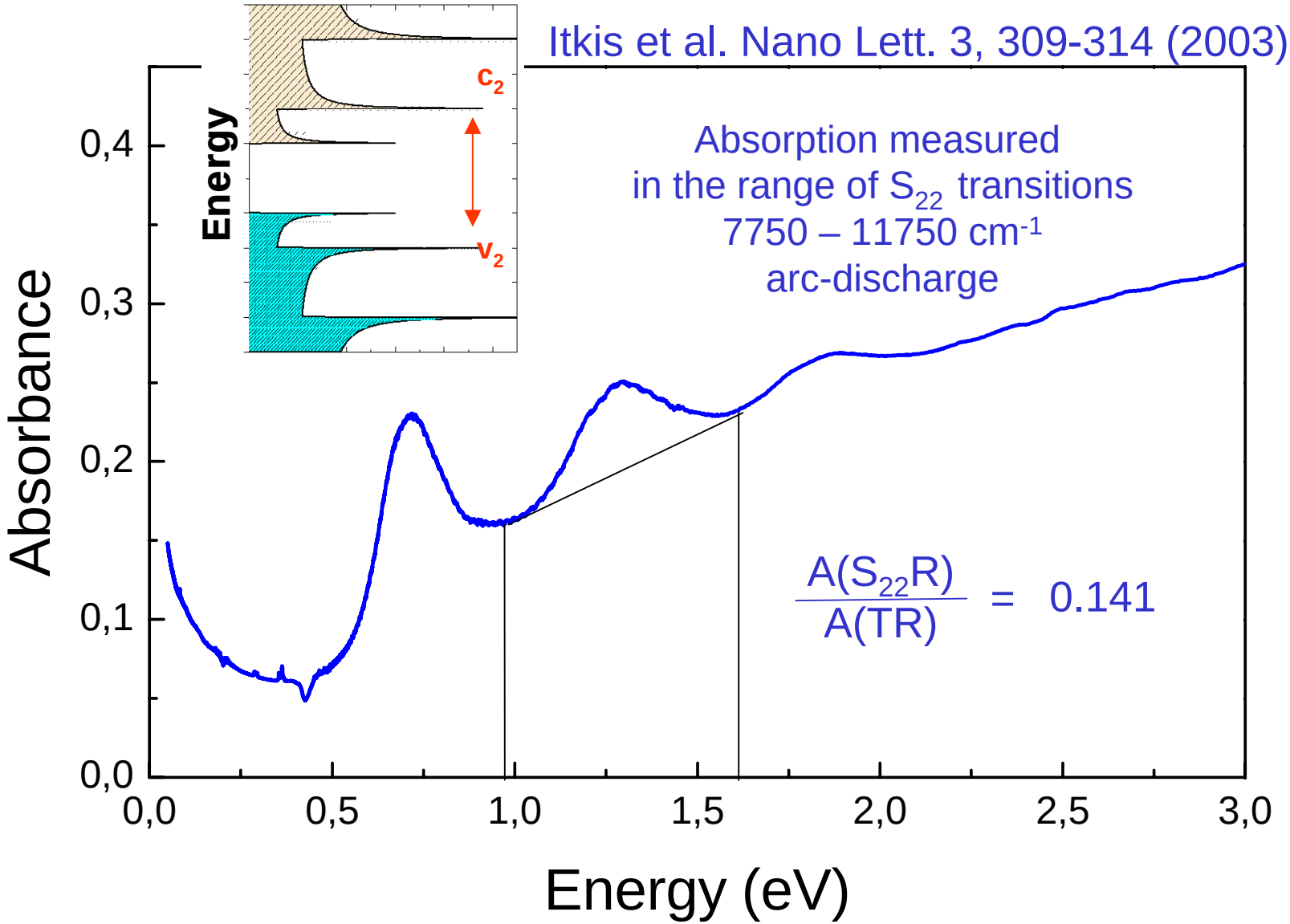
Outline

- Quantitative evaluation of SWNT purity by solution phase NIR spectroscopy
- Quantitative evaluation of graphitic impurities by XRD
- Quantitative evaluation of metallic impurities by Inductive Coupled Plasma (ICP)
- Electrical conductivity measurements of bucky paper by four leads method
- Materials investigated:
  - arc-discharge: Stuttgart, Shanghai, Montpellier
  - laser ablation: Karlsruhe, Ottawa

Protocol for quality control: homogenization

Charges: 10 - 50 g of soot mechanically homogenized until fine powder is obtained, 5 min at lowest speed stage

The procedure for quantitative analysis of NIR spectra:



Quantitative analysis of absorption: sample preparation

Itkis et al. Nano Lett. 3, 309-314 (2003)

50 mg of homogenized soot, dispersed in 100 ml of DMF by ultrasonic horn for 1 min, pulsed, 0.5 sec, 20 % of power,
UP 200S (max. 200 W)

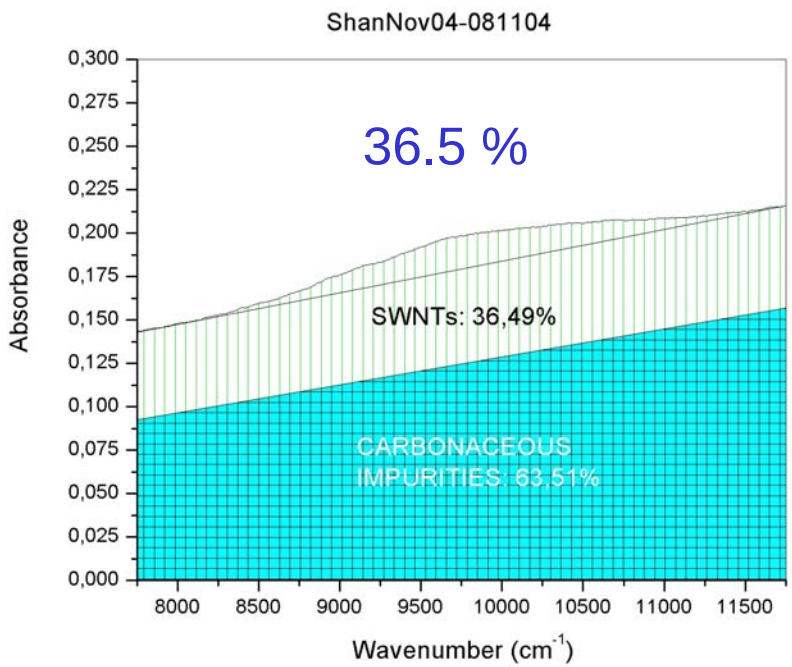
a few drops (3-7) of slurry diluted to 10 ml DMF,
must be a pale liquid without any light scatering particles !!!

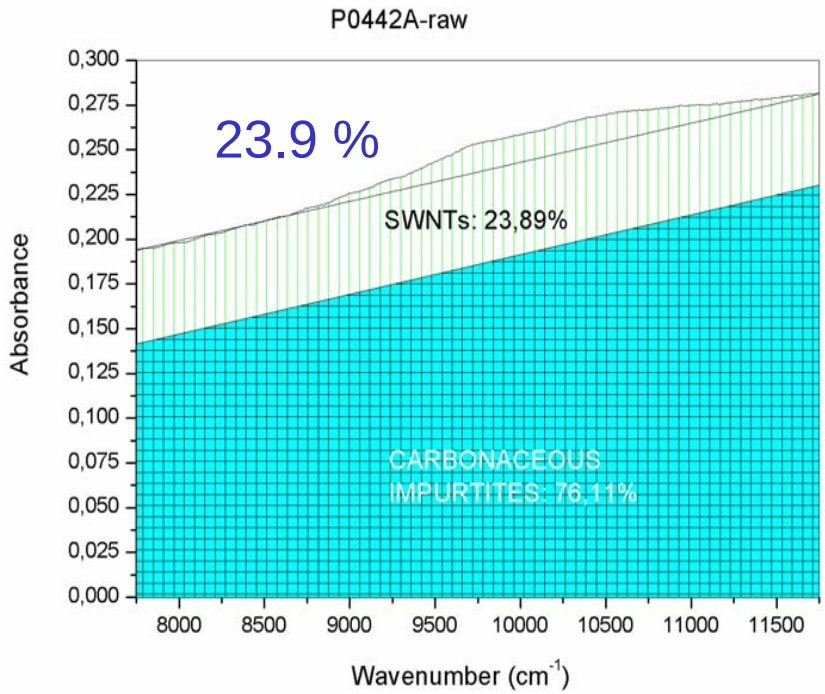
represent a purity of a large batch of 10 g SWNTs

Purity evaluation of as-prepared arc-discharge samples by use of a reference

$$P(S) = \frac{A(S_{22},S)/A(T,S)}{A(S_{22},R)/A(T,R)} \times 100\ \%$$

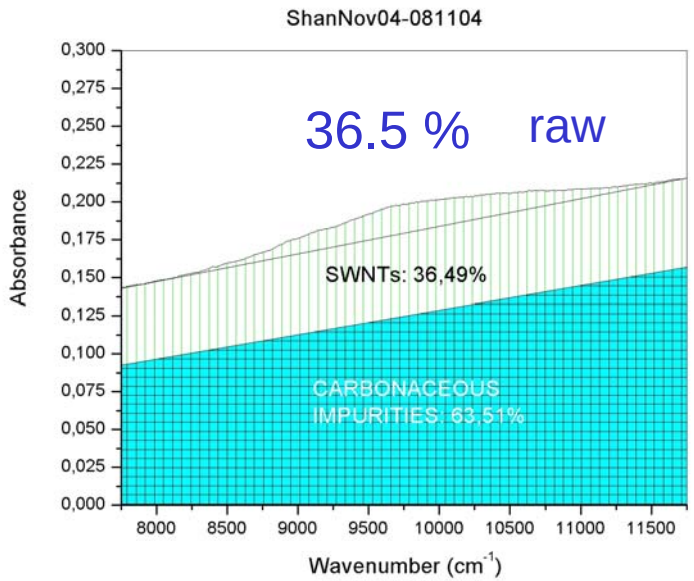
$A(S_{22},R)/A(T,R) = 0.141$

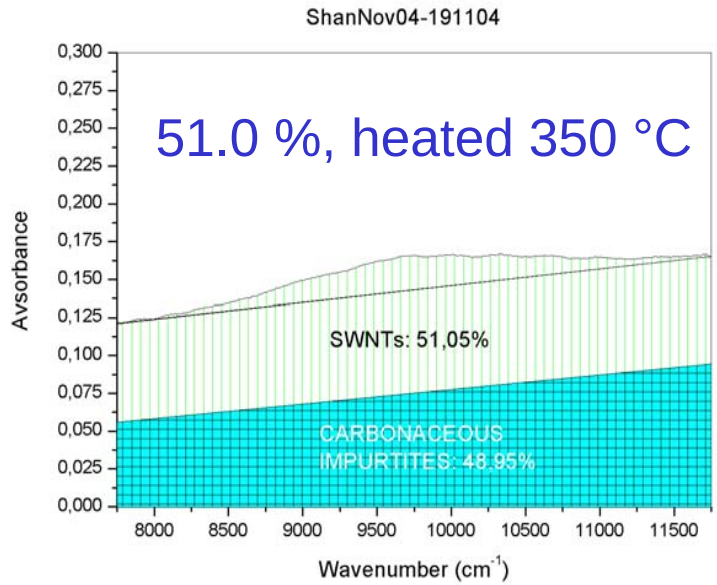


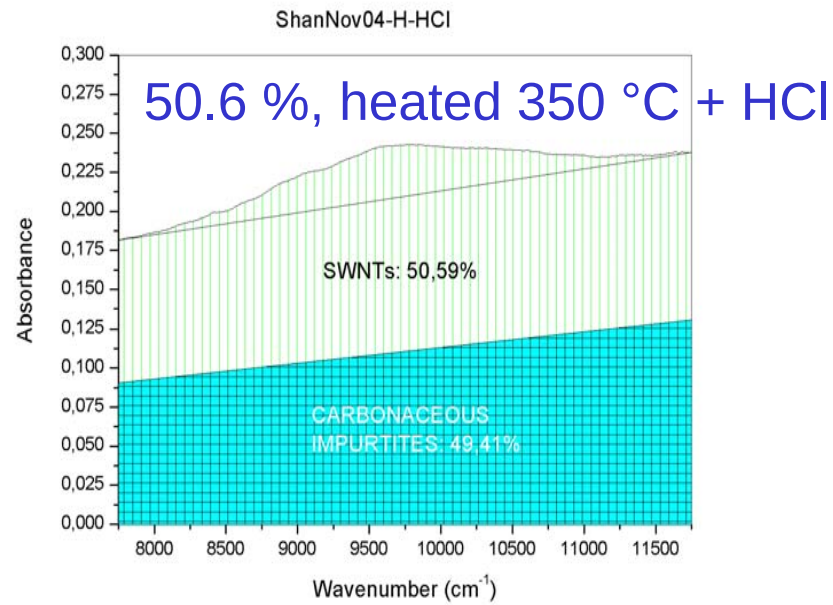


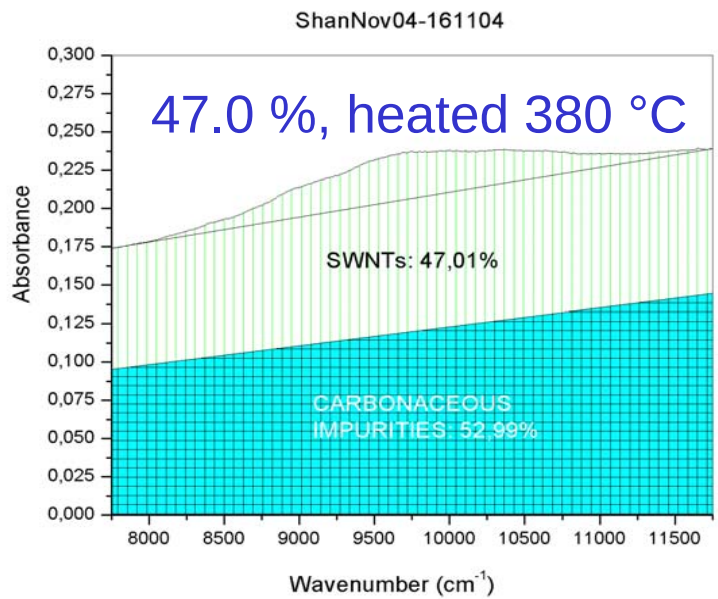
Sample	1	2	3	4	5	mean value (SD)
Relative purity, %	36.7	34.2	34.5	39.4	37.8	36.5 (± 3)

Quantitative evaluation of purification

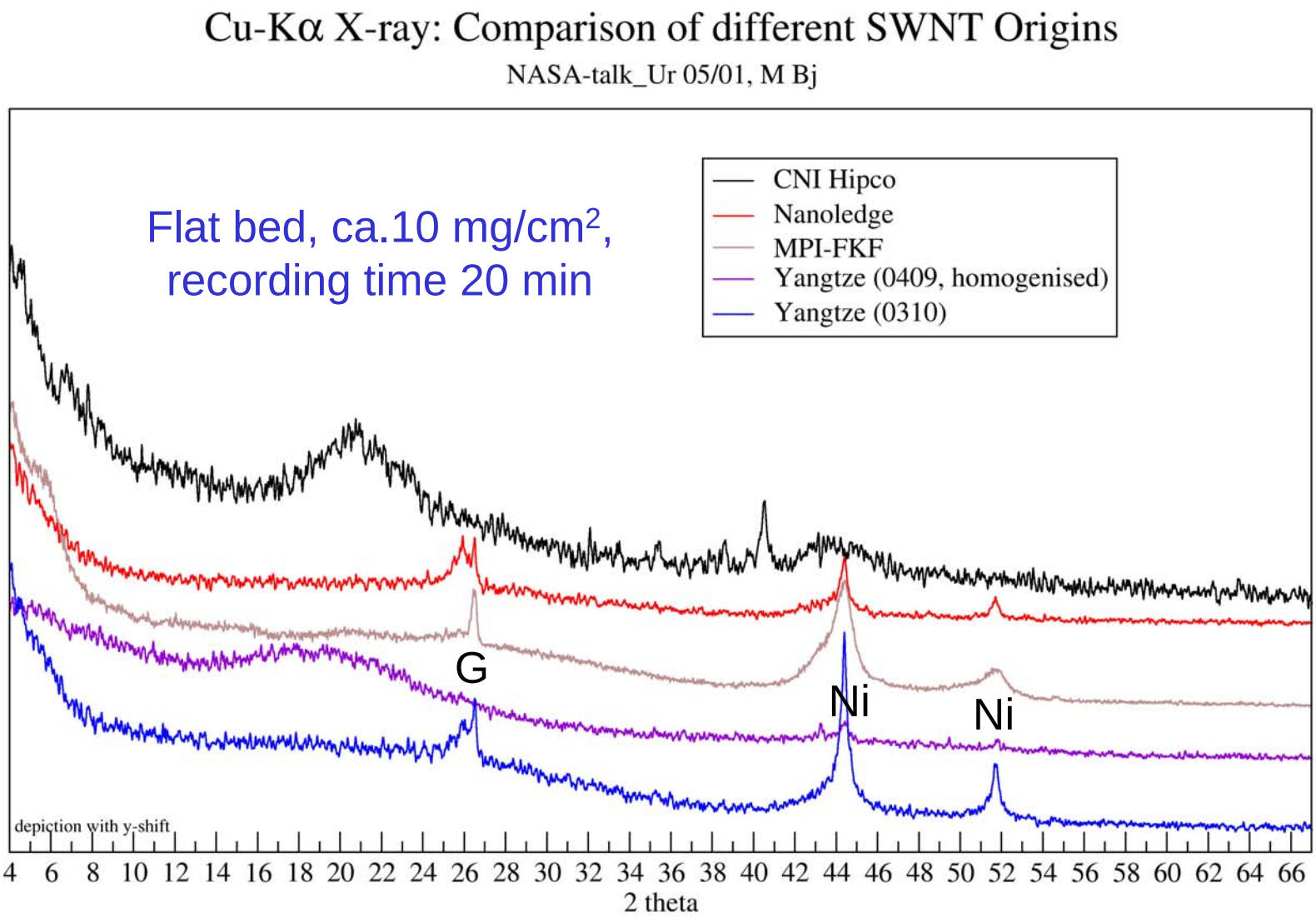








Powder X-ray Diffraction Analysis



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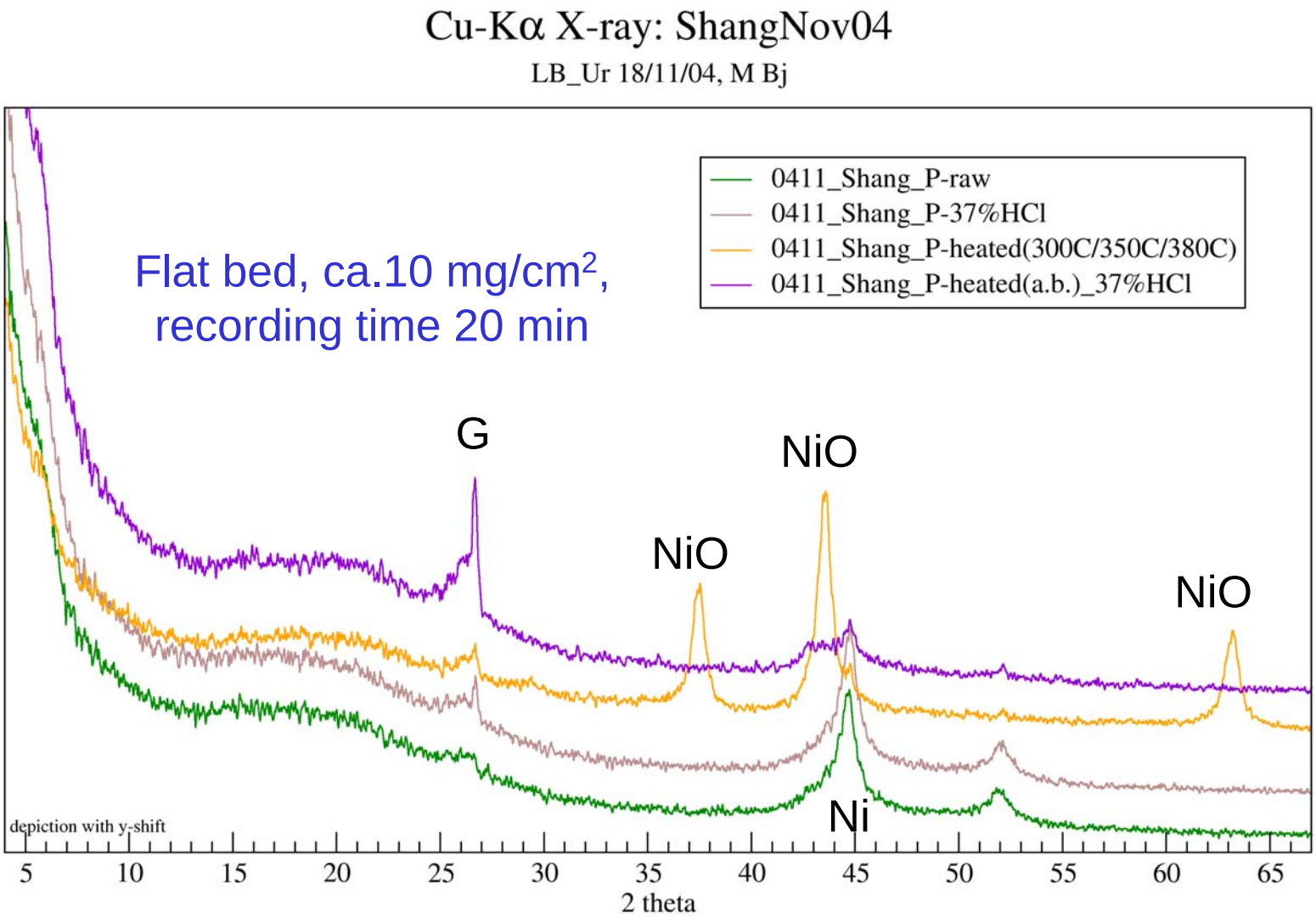
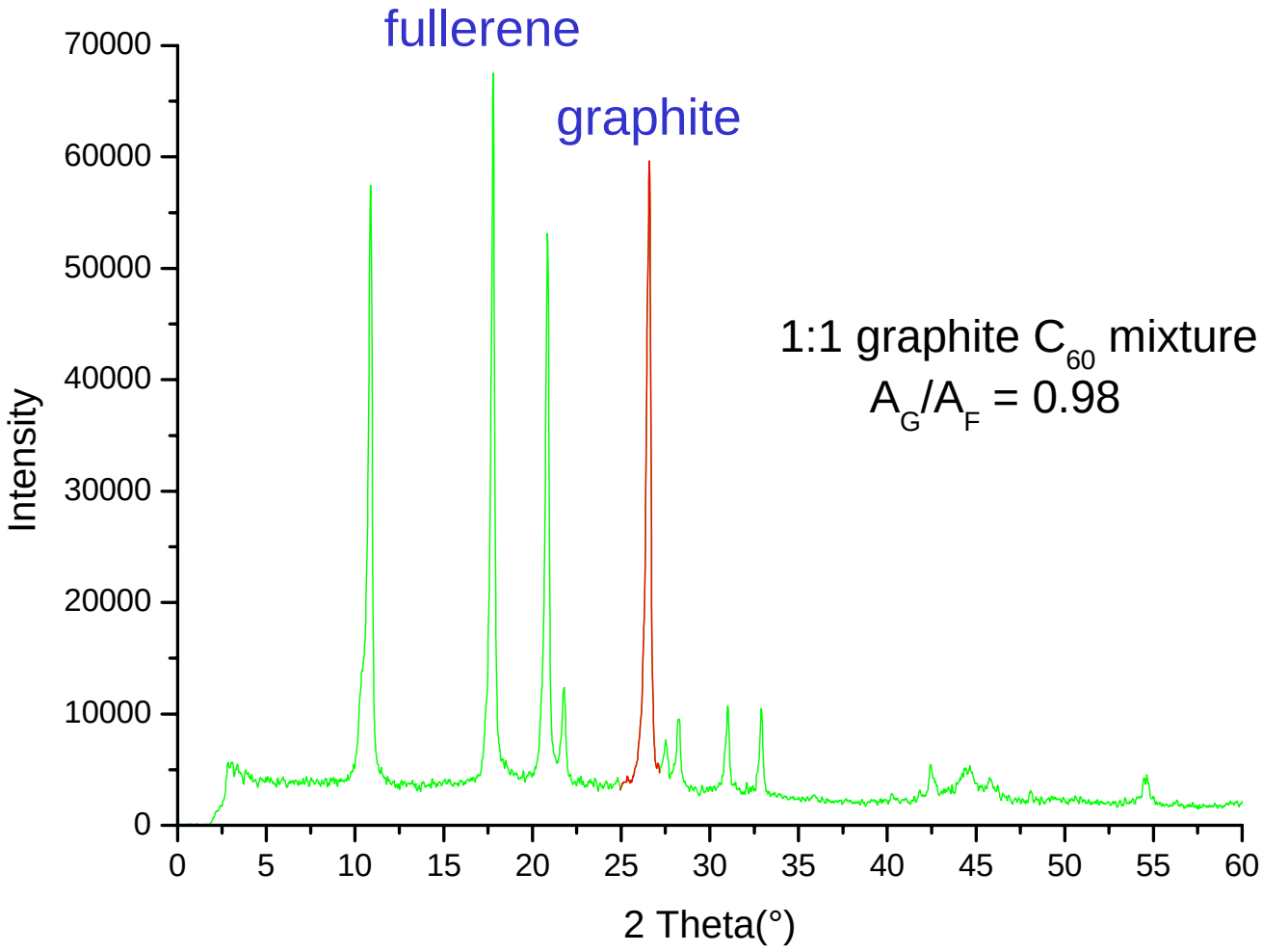
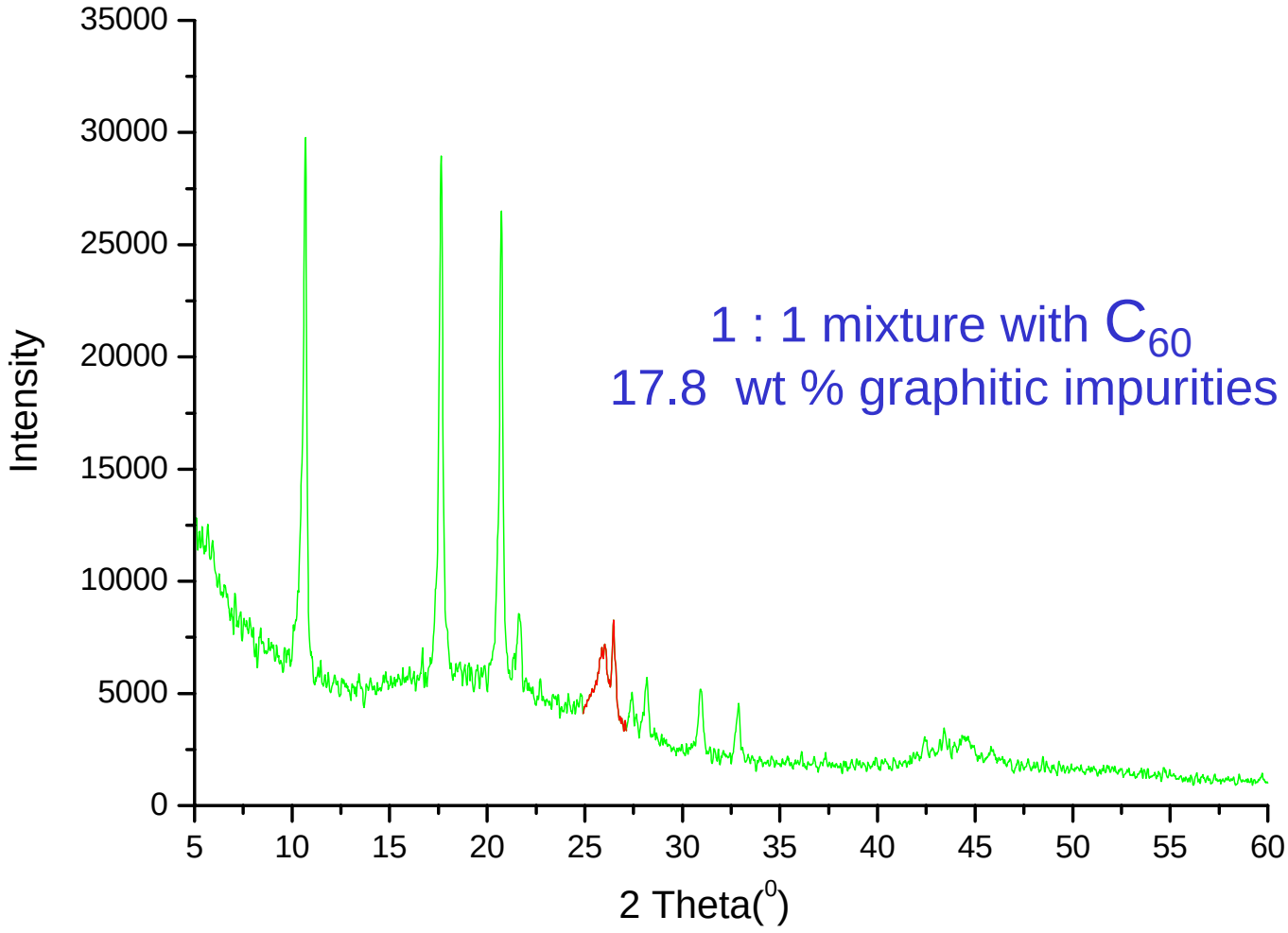


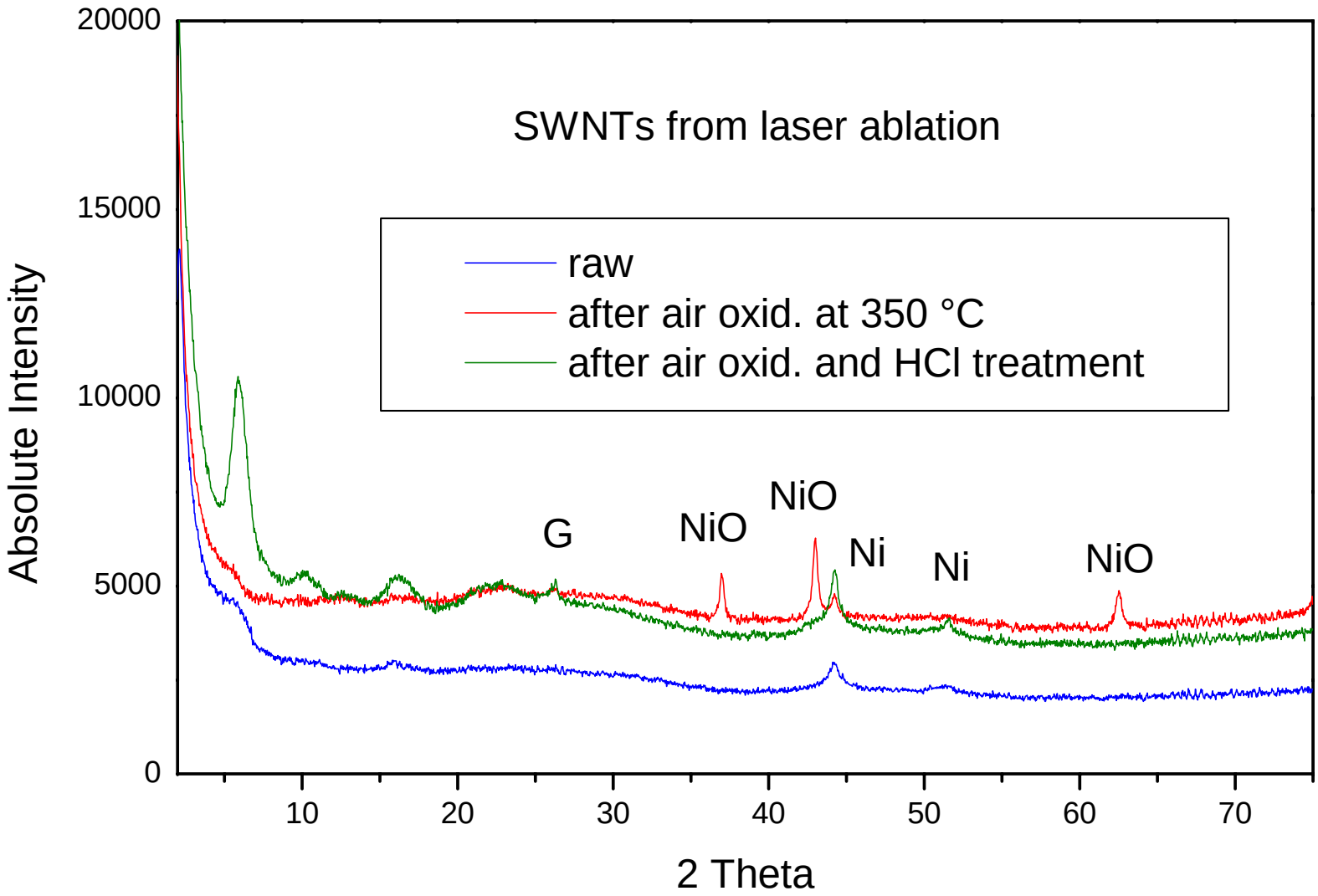
X-ray Diffraction of a 1:1 Mixture of C_{60} and Graphite



XRD evaluation of graphitic impurities



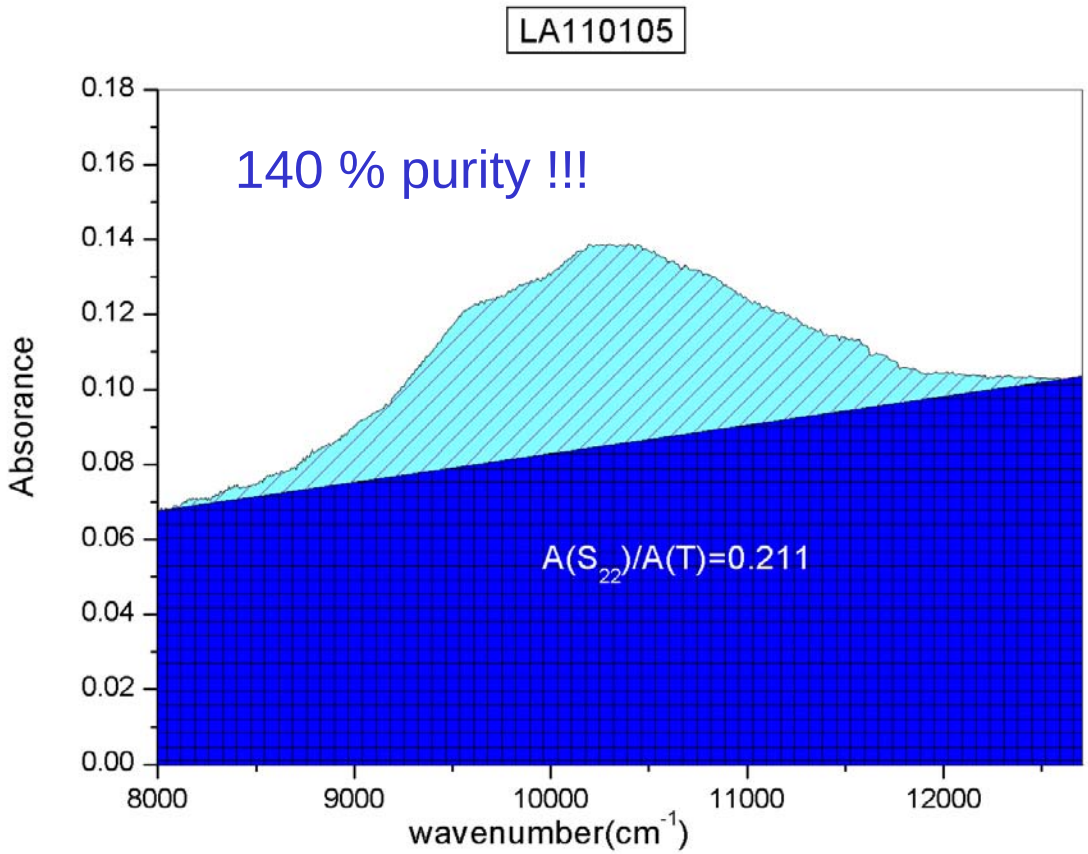
Purification of laser ablation sample



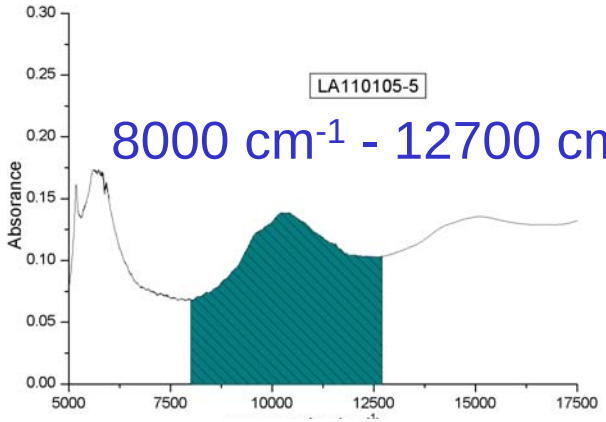
Quantitative purity evaluation of laser ablation samples

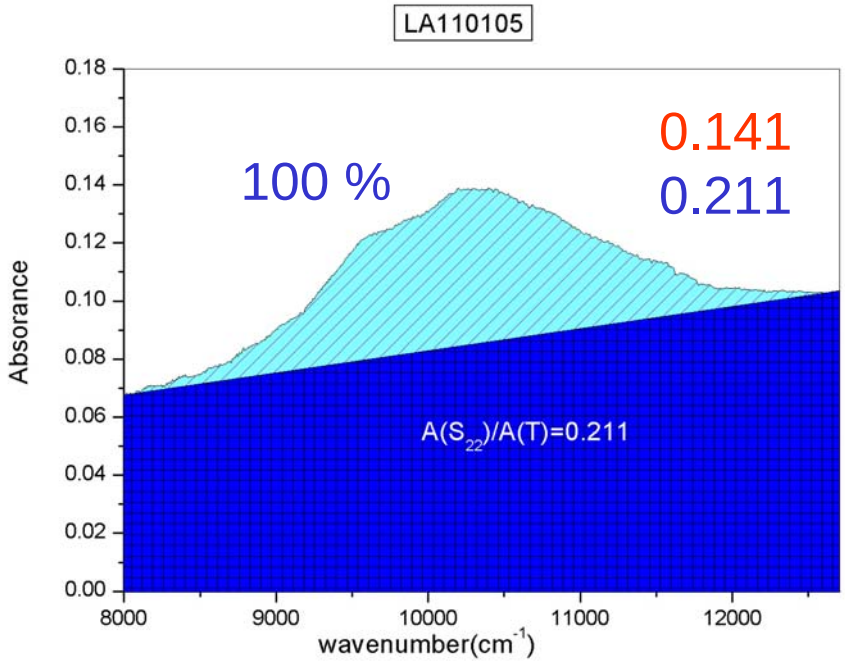
$$P(S) = \frac{A(S_{22},S)/A(T,S)}{A(S_{22},R)/A(T,R)} \times 100\ \%$$

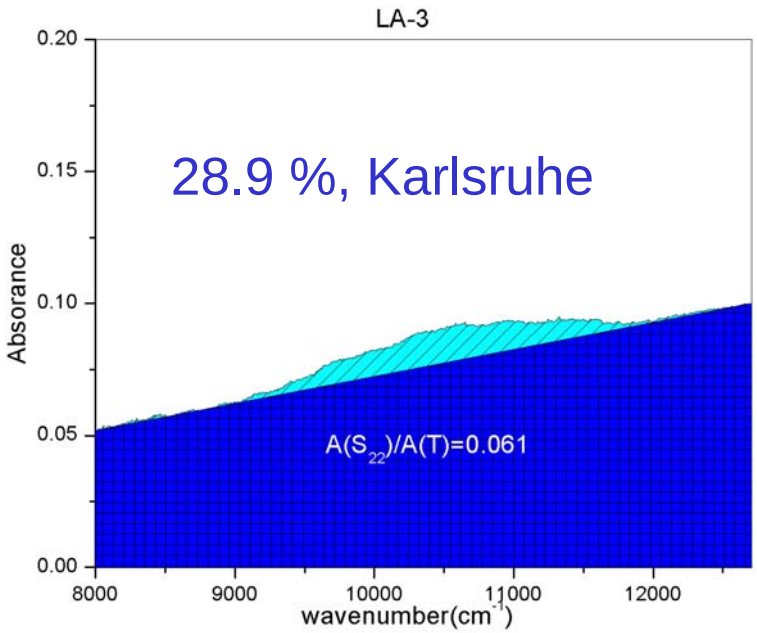
$A(S_{22},R)/A(T,R) = 0.141$

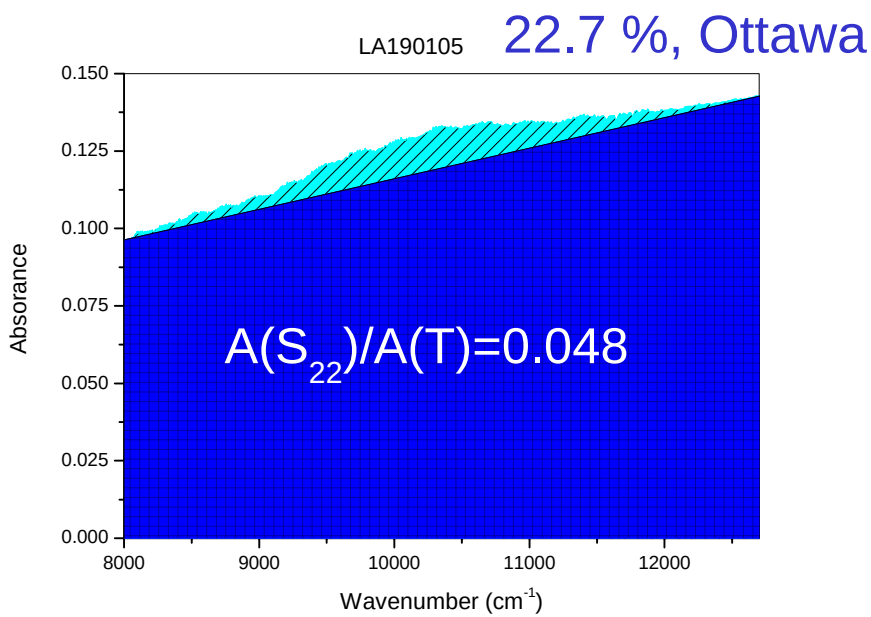


Quantitative purity evaluation of laser ablation as-prepared materials









Metal analysis by use of ICP

Elemental analysis of as delivered SWNT materials, wt %

Arc-discharge

Sample	Ni	Y
NL-1	20.7	2.2
Clem-1	19.0	4.7
Clem-2	15.8	3.3
NL-2	12.3	5.2
NL-3	4.0	2.4
Yang-1	1.7	3.8

Laser ablation

Sample	Ni	Co
K-4	10.4	9.9
CNI-1	3.5	3.5
CNI-2	1.2	3.0
K-5	1.6	1.6
K-10	0.8	0.9

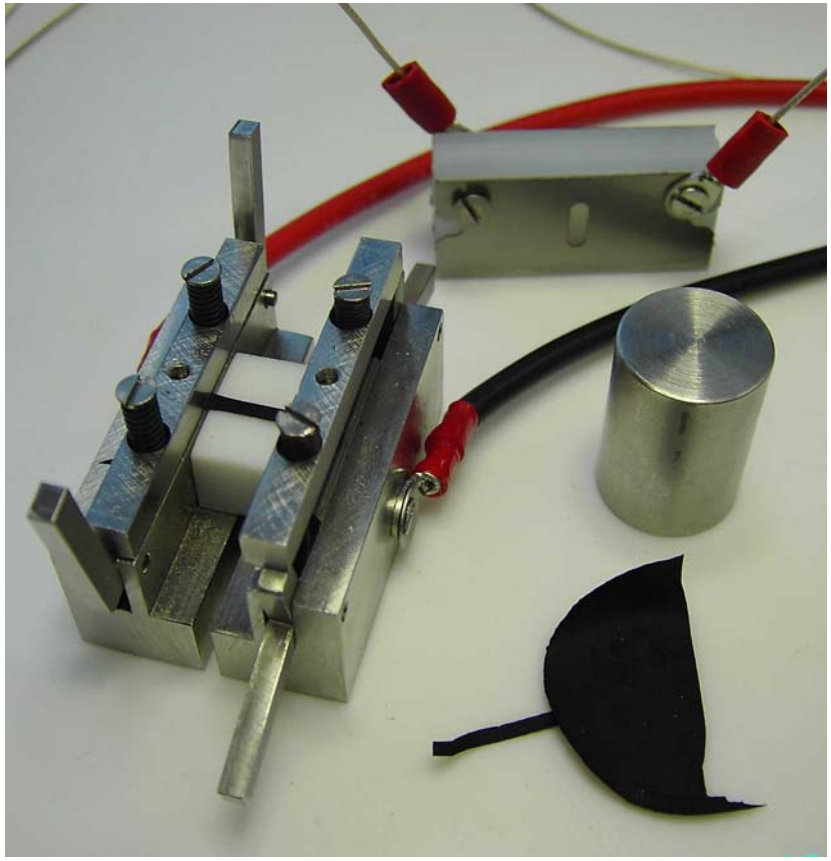
Hipco

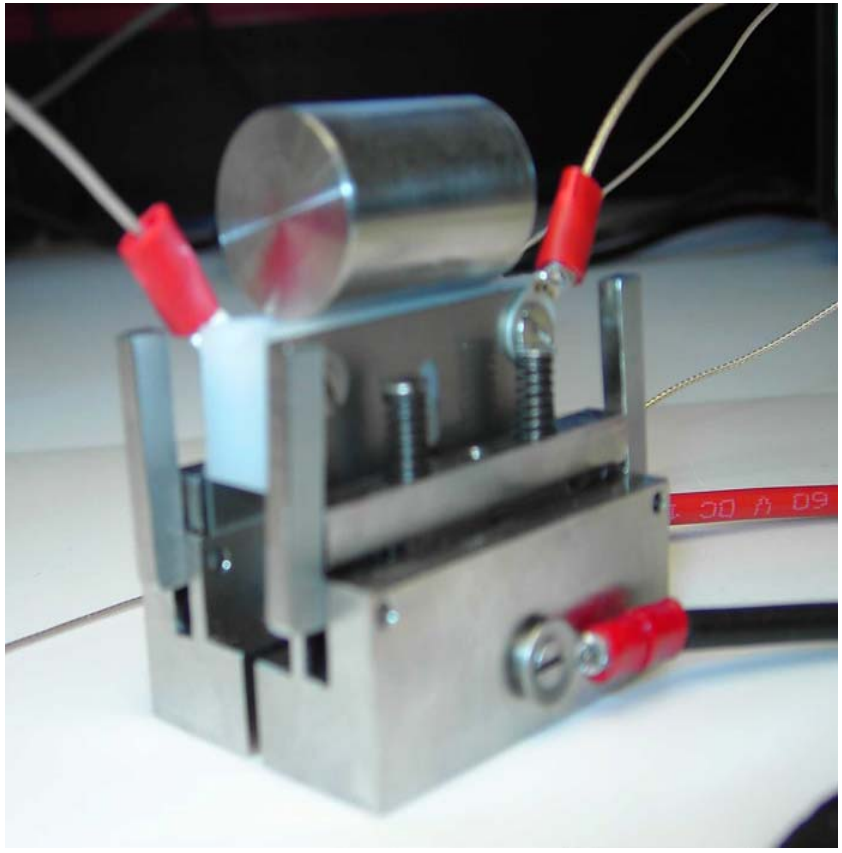
Sample	Fe
Hipco-1	25.0
Hipco-2	24.5
Hipco-6	16.4
Hipco-7	13.1
Hipco-3	6.5
Hipco-5	5.9

Sample preparation: 10 mg, 3 ml HCl, 3 drops HNO_3 , heated under pressure for 18h, emission spectra measured from solution

Electrical conductivity characterization of bucky papers

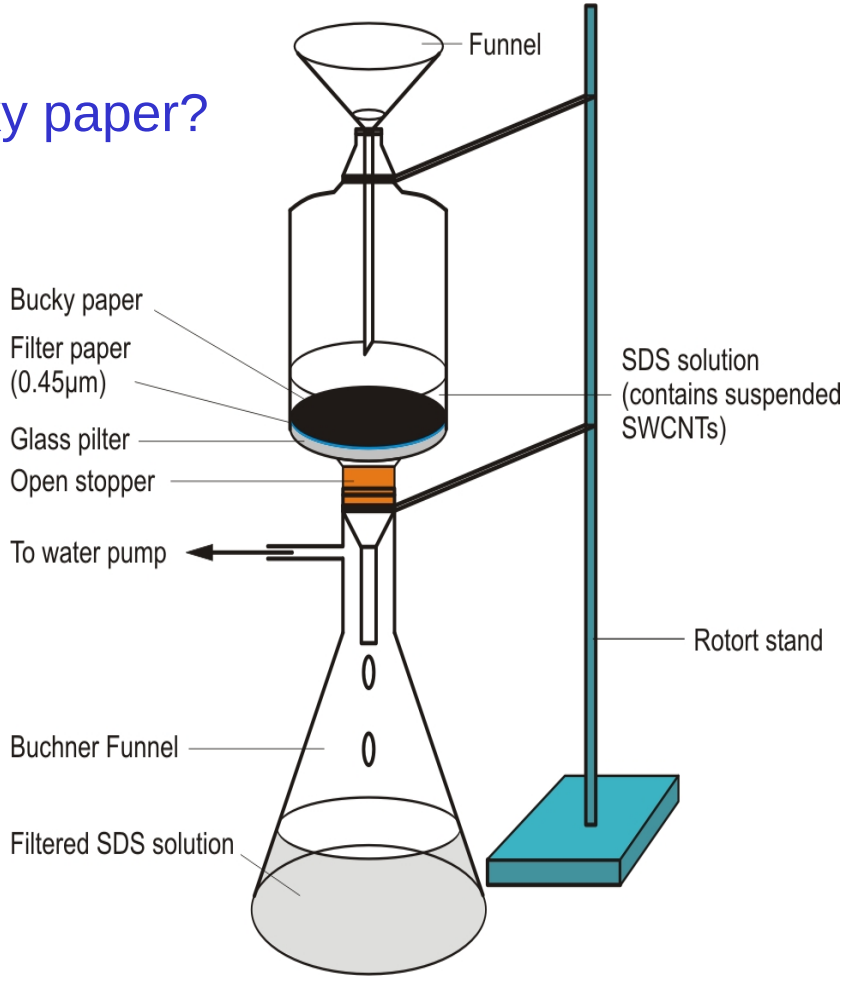
Standard device for conductivity measurement of bucky paper by four leads method





Electrical conductivity characterization of bucky paper

How to make a bucky paper?



Suspension of 1mg/ml in SDS prepared by ultrasonic agitation

Electrical conductivity characterization of bucky paper

Sample	relative purity %	Conductivity S/cm
Arc as-prepared	31.3	60 ± 6
Heated in air 350 °C, 20 min	36.0	140 ± 10
Heated in air 320°C, 40 min	39.3	140 ± 5

State-of-the-art in quality of carbon nanotube materials

News@nature.com

Published online: 10th December 2004

Nanotube suppliers accused of selling shoddy goods

Matthew Nordan, Lux Research, New York says:

„Researchers who buy products such as carbon nanotubes are **frequently being sold defective materials**."

"We heard one **horror story** after another".

"One semiconductor company found that a third of a sample of carbon nanotubes was actually **iron** left over from the production process".

"Materials ordered just weeks apart have widely different characteristics"

Conclusion: the situation is unlikely to change until standards for characterization are developed

Collaborators and Funding

- S. Roth
- J. Wang
- J. Liang
- B. Hornbostel

- FINANCIAL SUPPORT: European Projects:

- SPANG
- CANAPE
- CARDECOM